HEATING ELEMENT EMBEDDED PARYLENE MICROCOLUMN FOR MINIATURE GAS CHROMATOGRAPH

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ABSTRACT

This paper presents the first miniature parylene gas chromatograph (GC) column with embedded heating element. The main advantage of a parylene microcolumn is its lower heat capacity compared with a micromachined silicon/glass column. The analysis time and power consumption of the miniature GC system can be decreased with a parylene microcolumn because of its rapid transient heating. In this paper, the fabrication and heat transfer of a heating element embedded parylene microcolumn is presented.

INTRODUCTION

A miniature, integrated chemical laboratory (µChemLab) has been developed at Sandia National Laboratories for the detection of chemical warfare agents or explosives [1]. The main requirements for this application are trace detection (ppb level) in "real-world" environment and rapid analysis to provide early warning. These requirements are closely related with GC column efficiency. While conventional GC systems use a fused silica capillary column, miniature GC has utilized micromachined silicon channels that can be anodically bonded to glass plate because this micromachined silicon/ glass column can provide improved ruggedness, smaller size, lower power consumption for heating and an ability to integrate other components on a same chip [2-4]. Recently, there has been progress in the fabrication and simulation of a deep and narrow silicon channels using deep reactive ion etching (DRIE) [1, 5, 6]. This deep and narrow micromachined silicon channel can decrease the required column length because the geometry helps gases react with the stationary material which is coated inside the column, and consequently it leads to a shorter analysis time. Also, sample detention time decreases as column temperature increases. It has been reported that gas separation can be achieved in less than 30 seconds at a higher column temperature (80°C) [1]. Therefore a miniature column which can be heated with low power is the key to achieving a rapid analysis.

Parylene (poly paraxylylene) is a candidate for GC column material because of its chemical inertness, low permeability and low heat capacity [7]. In addition, a parylene corrugated membrane such as open channel can be fabricated simply by coating a micromachined silicon channel. Recently, some free-standing parylene structures such as microvalve have been fabricated by using a photoresist as sacrificial material [8]. However this method has limitations when fabricating very long and high structures such as GC columns. An enclosed parylene column can be formed by coating and fusion bonding between parylene membranes. The details of this process have been described elsewhere [9].

FABRICATION OF PARYLENE COLUMN

The first step in fabrication of a parylene column is the etching of a silicon microchannel mold. Arrays of spiral microchannels (100µm wide, 350µm deep, 1m long) are etched on 4" diameter silicon substrates using DRIE. Figure 1 is a SEM profile of the cross-section of a micromachined silicon channel. The array of silicon microchannels are cut into single die, 2cm by 2cm square. The channel ends are wider (400µm) and also extend to the edges of each die.

Figure 2 shows the overall fabrication process flow. The silicon microchannel is coated with 10µm thick parylene. 'Parylene C' (Poly mono-chloro-para-xylylene ; *Special Coating System*; Indianapolis, IN) is used in this research. A Pyrex glass plate which has the same area as silicon microchannel is also coated with 10µm thick parylene. Parylene/parylene bonding is achieved in vacuum oven under the condition of 200°C and 24MPa applied pressure.

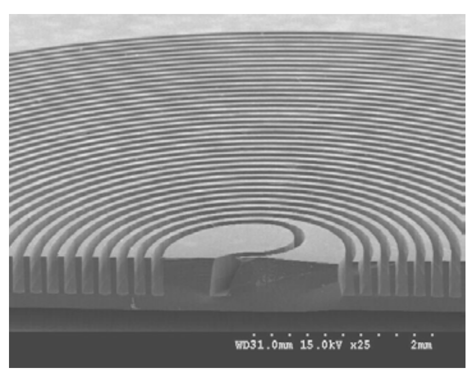

Figure 1. A micromachined silicon channel.

The bond process takes place under high temperature and pressure where polymer chains at the interface of two parylene membranes can be rearranged and entangled resulting in fusion.

After bonding, a free-standing parylene column can be obtained by KOH etching (20% KOH, 5hrs at 80°C) of the silicon microchannel. A transparent free-standing parylene column is shown in Figure 3. In general top/ bottom tubing connection for micromachined silicon/glass column cannot be used for a parylene microcolumn because it is a flexible polymer column. Instead, parylene column was designed to have side connection as shown Figure 3. Polyimide coated silica microtubes (OD ~ 350µm, ID ~ 100µm square, *Polymicro*, Phoenix, AZ) are inserted into tapered ends of the parylene microcolumn and then sealed with epoxy adhesive.

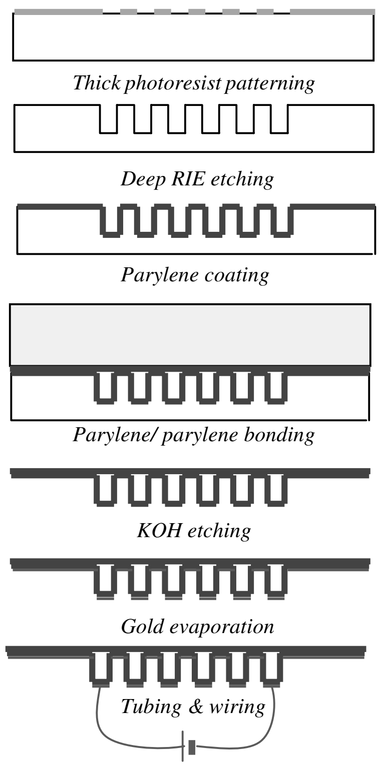

Figure 2. Fabrication process flow for heating element embedded parylene microcolumn.

The heating element can be fabricated simply by electron beam evaporation of gold onto the corrugated surface of the parylene column. Since the parylene column has a rectangular geometry, gold is deposited only on the top and the bottom surfaces, not on the wall of parylene column. The gold thin film deposited on the top of parylene column forms a 1m long wire along the parylene column. Joule heating ($P=V^2/R$) can be generated by applying voltage to this thin gold film. The resistance of the gold wire depends on its thickness. However, it was found that if more than 0.2µm of gold is deposited, the wall of parylene column can also be slightly coated with gold, resulting in a short circuit. In order to avoid this effect, 0.15µm of gold was deposited.

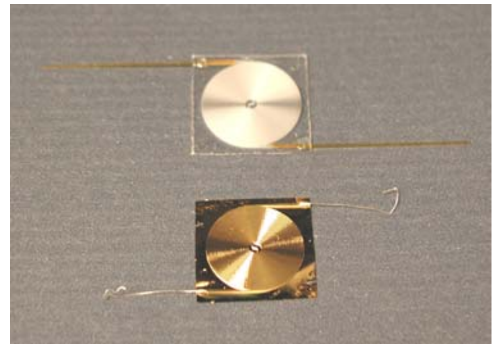

Figure 3. Free-standing parylene column (top) and gold deposited parylene column (bottom)

PRESSURE DROP IN PARYLENE COLUMN

To evaluate the parylene/parylene bond integrity, a column was pressurized by flowing water through with a pump. No inter-channel leakage was found in this test. Pressure drop was measured at different flow rates using nitrogen gas. A differential pressure sensor (max. ~ 1000Torr, *MKS Instruments*, MA) and a flow controller (max. ~ 1sccm, *MKS Instruments*, MA) were used for this measurement. The measured values shown in Table 1 have good agreement with the calculated values assuming Poiseuille flow. 3-D ANSYS simulation was carried out in order to find out the velocity entrance length, and the result was less than 1mm. Therefore, the nitrogen flow can be considered a fully developed flow because the entrance length is much shorter than column length, 1m.

Flow rate (sccm)	0.2	0.4	0.6	0.8	1.0
Measured ΔP (Torr)	25	49	70	89	109
Calculated ΔP (Torr)	20	41	61	81	102

Table 1. Flow rate versus pressure drop in parylene microcolumn.

HEATING OF PARYLENE COLUMN

The thermal characteristics of interest for this parylene column with embedded heating element are, how fast can it be heated and how uniform is the temperature.

Heat transfer analysis of a parylene microcolumn was carried out using ANSYS simulation. First of all, a 3D analysis is used to find out the entrance length where the temperature of nitrogen is fully developed. The entrance length was negligible (less than 1mm) compared to column length (1m). Hence a simplified 2D analysis was used to obtain transient temperature change, and temperature distribution in the cross-section of the column. Free convection heat transfer is considered for each surface of the parylene column, and temperature dependant convection heat coefficients are used for this analysis. However, some empirical data are required to find out the convection coefficients for parylene microcolumn as shown in Figure 4. An Infrared camera (thermaCAM PM190, *Inframetrics*) was used to measure the temperature of parylene column.

Figure 5 shows the ANSYS results for the steady-state temperature with applied power at different convection heat coefficients. The graph A is the result when the convection coefficient of the top surface (h_{top}) is fixed at 4 $W/m^2 \cdot C$ and the graph B is when the convection coefficient of the wall (h_{wall}) is fixed at 2.5 $W/m^2 \cdot C$. Since the bottom of parylene column was insulated with polystyrene foam when the temperature was measured (see Figure 4), the convection coefficient of the bottom was considered zero for each case. As shown in these graphs, the steady-state temperature of parylene column is more sensitive to h_{wall} than h_{top}. A good agreement with measured data is obtained when h_{top}= 4 $W/m^2 \cdot C$ and h_{wall} = 2.5 $W/m^2 \cdot C$. These convection coefficients are applied to the transient analysis. The time required to reach a steady-state, is less than 30 seconds for both ANSYS, and measurement. Accordingly, the parylene microcolumn can be heated to 100°C in 30 seconds with only 45mW. This is much less power consumption than micromachined silicon/ glass column (2°C/sec with 1.5W, 20°C/sec with 10W).

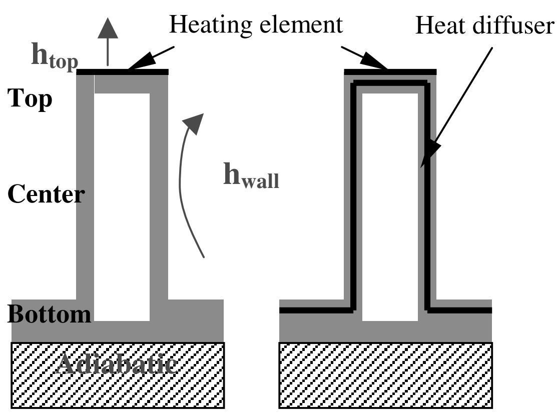

Figure 4. Cross-section of parylene columns.

Figure 6 is an example of temperature distribution taken by infrared camera across an area 1cm by 1cm of parylene column insulated on the base. The top surface of parylene column is focused in this picture. Along the path indicated AB, a uniform temperature distribution was observed. In other words, there is no temperature difference observed along the column radius.

However, the temperature difference in column cross-section cannot be easily measured with the infrared camera. Therefore, ANSYS simulation was used to estimate this effect. Figure 7 is a transient temperature profile at the top, center and bottom of the parylene column, as shown in Figure 4 (left). The temperature difference between top and bottom is about 5°C, which is significant.

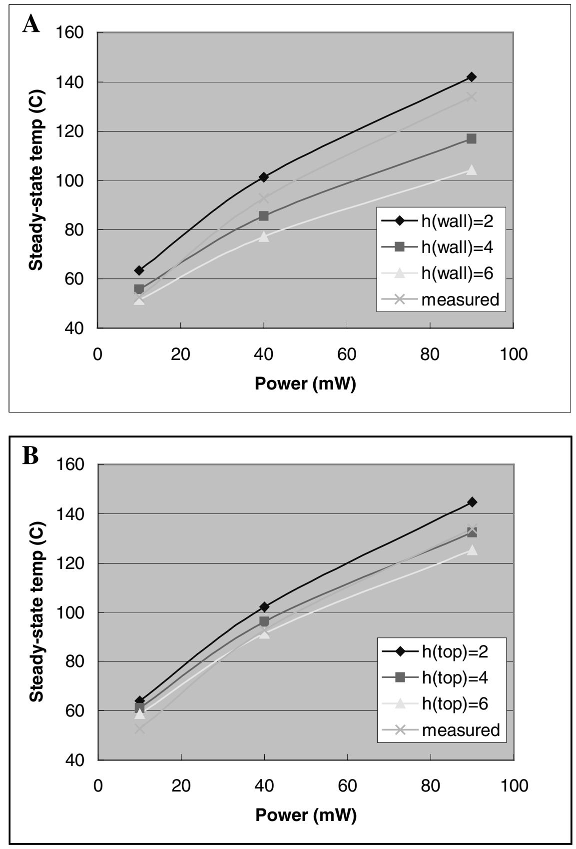

Figure 5. Steady-state temperature data for different applied power with convection heat coefficients($W/m^2 \cdot C$) as a parameter.

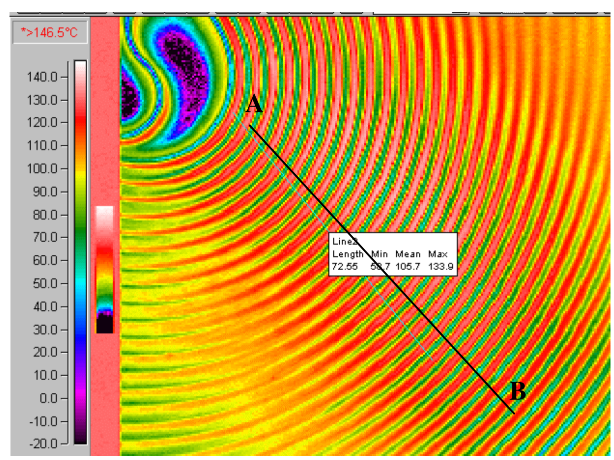

Figure 6. Temperature map when 90mW is applied.

To reduce this temperature difference across the column cross-section, a triple layer column has been designed and its heat transfer investigated. A thin metal layer is inserted between two parylene layers as a heat diffuser (Figure 4 right).

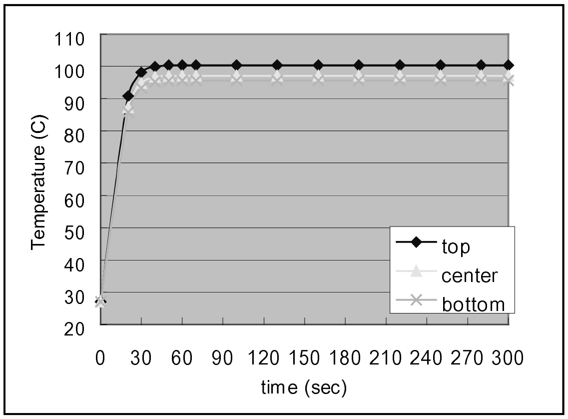

Figure 7. Transient temperature profile at different locations.

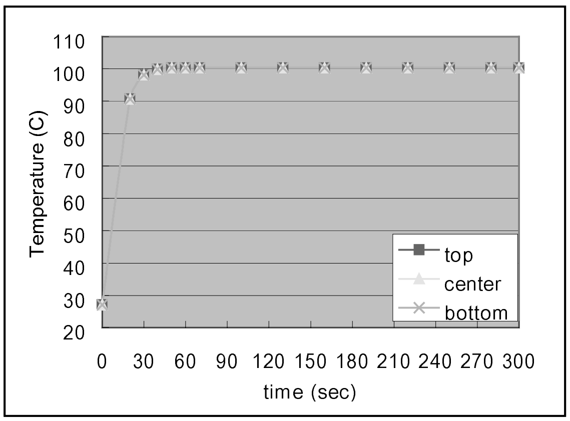

Figure 8. Transient temperature profile at different locations for triple layer parylene column.

The fabrication of this triple layer structure (5µm parylene/ 0.1µm Al / 5µm parylene) was presented in our previous paper [9]. ANSYS result in Figure 8 shows that the insertion of a heat diffuser is very effective to achieve a uniform temperature distribution in the column cross-section. The temperature difference was less than 1°C.

CONCLUSIONS

A 1m long parylene microcolumn with rectangular cross-section (100µm wide, 350µm high) for a miniature GC was fabricated. The parylene column can reach the steady-state temperature of 100°C in 30 seconds only with 45mW using the embedded heating element. Temperature measurement with an infrared camera showed a good interchannel temperature uniformity. Temperature difference in parylene column cross-section can be reduced into less than 1°C by forming a triple layer structure, which contains a thin metal diffuser between the parylene layers.

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